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STUDIES OF LAKE MICHIGAN BOTTOM SEDIMENTS—NUMBER SEVEN

DISTRIBUTION OF MERCURY IN UNCONSOLIDATED SEDIMENTS FROM SOUTHERN LAKE MICHIGAN

E. Joyce Kennedy, R. R. Ruch, and N. F. Shimp

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ABSTRACT

The mercury content of 132 unconsolidated sediment samples from 31 sampling stations in southern Lake Michigan was determined by a radiochemical separation technique. In the deep central portion of the southern part of the lake and in areas west of Benton Harbor, Michigan, and southwest of Grand Haven, Michigan, mercury has accumulated (0.1 to 0.4 part per million) in the uppermost sediment layers. A base level of 0.03 to 0.06 ppm mercury is found at depth in the cores.

In sandy areas along the southern and southwestern shores no accumulation of mercury is apparent near the surface of the sediments; an accumulation at a lower interval in these areas is believed to be associated with the presence of metal sulfides.

Mercury concentrations in the sediment-water interface samples evidently vary with the organic carbon and total sulfur concentrations of the samples.

INTRODUCTION

This is the seventh in a series of reports presenting results of an investigation of southern Lake Michigan sediments. Preceding reports in the

series (Shimp, Leland, and White, 1970; Ruch, Kennedy, and Shimp, 1970; Schleicher and Kuhn, 1970; Shimp et al., 1971) gave data on the concentrations of several major, minor, and trace elements in these sediments and discussed possible causes and interrelationships of the distributions found.

Because of great concern by the public and various levels of government regarding environmental pollution by mercury in several of the United States and provinces of Canada, there is an immediate need for data on mercury levels at specific locations in the environment. This report presents such data for the sediments of southern Lake Michigan.

Instances of mercury pollution in the Great Lakes region have recently been reported. Because of contamination by mercury discharges from chlor-alkali industrial plants on the shores of the St. Clair and Detroit Rivers, commercial fishing was banned in waters of the St. Clair River - Lake Erie system downstream from those plants in March (by Canada) and May (by the United States) of 1970 (Chem. and Eng. News, 1970a). Mercury levels found in walleyed pike from Lake Erie and the St. Clair River were as high as 3.6 ppm. The levels of mercury found in the sediments ranged from 86.0 ppm in a part of the Detroit River very close to a chlor-alkali plant to 0.4 ppm and less in the Detroit River near its mouth at Lake Erie.

Fishing was also banned in the Wisconsin River downstream from a chlor-alkali plant at Port Edwards, Wisconsin (Chem. and Eng. News, 1970b). Fish in the stretch of river affected contained as much as 1.13 ppm mercury. Sediments from the river bed near the plant showed as much as 560 ppm mercury, while 4 miles downstream the mercury levels in the sediments were still as high as 50 ppm.

Investigation of fish in a fish kill in June, 1970, in the Iron River at Caspian, Michigan, revealed that they contained up to 2.3 ppm mercury (Turney, 1970). The source of the mercury pollution was believed to be phenylmercuric acetate used as a mildew inhibitor by a laundry, which had been discharging 0.2 lb of mercury a day into the river for several years.

Mercury and most of its salts are water insoluble, but once deposited in lake and river sediments they do not necessarily remain isolated from the lake or river ecosystem. The oxidation of elemental mercury to the mercuric ion occurs in an aerobic lake when organic complexing agents are present. Jensen and Jernelov (1969) demonstrated that microorganisms living in the sediments ingest the mercuric ion and transform it into dimethylmercury or the methylmercuric ion. Most of the volatile dimethylmercury moves rapidly through a body of water into the atmosphere, from whence it might be carried to land. The methylmercuric ion and some dimethylmercury are incorporated into plant and animal tissue. As one kind of fish is preyed upon by another higher in the food chain, the mercury accumulates in the predator fish eaten by man. Data on mercury levels in the sediments of lakes that provide food resources for man are thus of great importance.

Preparation of Samples

A total of 132 unconsolidated sediment samples from 31 sampling stations (fig. 1) in southern Lake Michigan were analyzed in this study. Table 1 gives the exact location of each station, the water depth, and the type of sample tested.

Staff members of the Stratigraphy and Areal Geology Section of the Illinois State Geological Survey were in charge of the collection and storage of the sediment cores and grab samples, as described by Gross et al. (1970). Excess water was siphoned off the top of the cores immediately after sampling. The 3-inch cores were sealed in their plastic liners and frozen aboard ship. Grab samples were immediately sub-sampled and the sub-samples placed in plastic bags and frozen. The 2-inch cores were sealed in plastic liners, stored in an upright position aboard ship, and transferred to a 5° C storage compartment at the laboratory in Urbana. All 3-inch cores and grab samples were kept frozen and the 2-inch cores kept at 5° C until prepared for analysis to prevent mixing and to hinder the biological activity in the sediment. The cores were extruded from their liners and cleaved into sections, which were washed with deionized water and then thawed and dried. The frozen grab samples were removed from the plastic bags and air dried. Such minimal handling of the sediment samples while they were frozen is believed to have prevented mixing and contamination that would have been easily apparent in trace metal analysis.

Preliminary analyses were done in triplicate of (a) air-dried, (b) freeze-dried, and (c) oven-dried (110° C) fractions of the top interval of sediment from core 210. The air-dried samples gave an average value of 0.12 ppm mercury, the results on the freeze-dried samples were 23 percent lower, and the results for the oven-dried samples averaged 7 percent lower than the results for the air-dried samples.

The mercury lost in the oven-dried and freeze-dried samples would actually be greater than that calculated because the sediment samples contain moisture,and loss of moisture on oven drying or freeze drying would partially compensate for mercury losses in the samples so treated. When the moisture loss is considered, the mercury loss from the oven-dried samples is estimated to be in the range of 8 to 11 percent.

The sediment samples from cores (101 through 140) taken early in the Lake Michigan program had all been oven dried at 110° C. A portion of each sample from the more recent corings was set aside for air drying, exclusively for purposes of mercury analysis. However, since the results of mercury analysis on oven-dried samples are evidently not lower than those on air-dried samples by a factor greater than the precision of the method, we believe that no erroneous conclusions have been drawn by comparing results from oven-dried and air-dried core samples.

METHODS

Determination of Mercury by Neutron Activation Analysis with Radiochemical Separation

Weighed sediment samples of approximately 1 gram each were placed in half-dram polyethylene snap-cap vials and irradiated in the University of

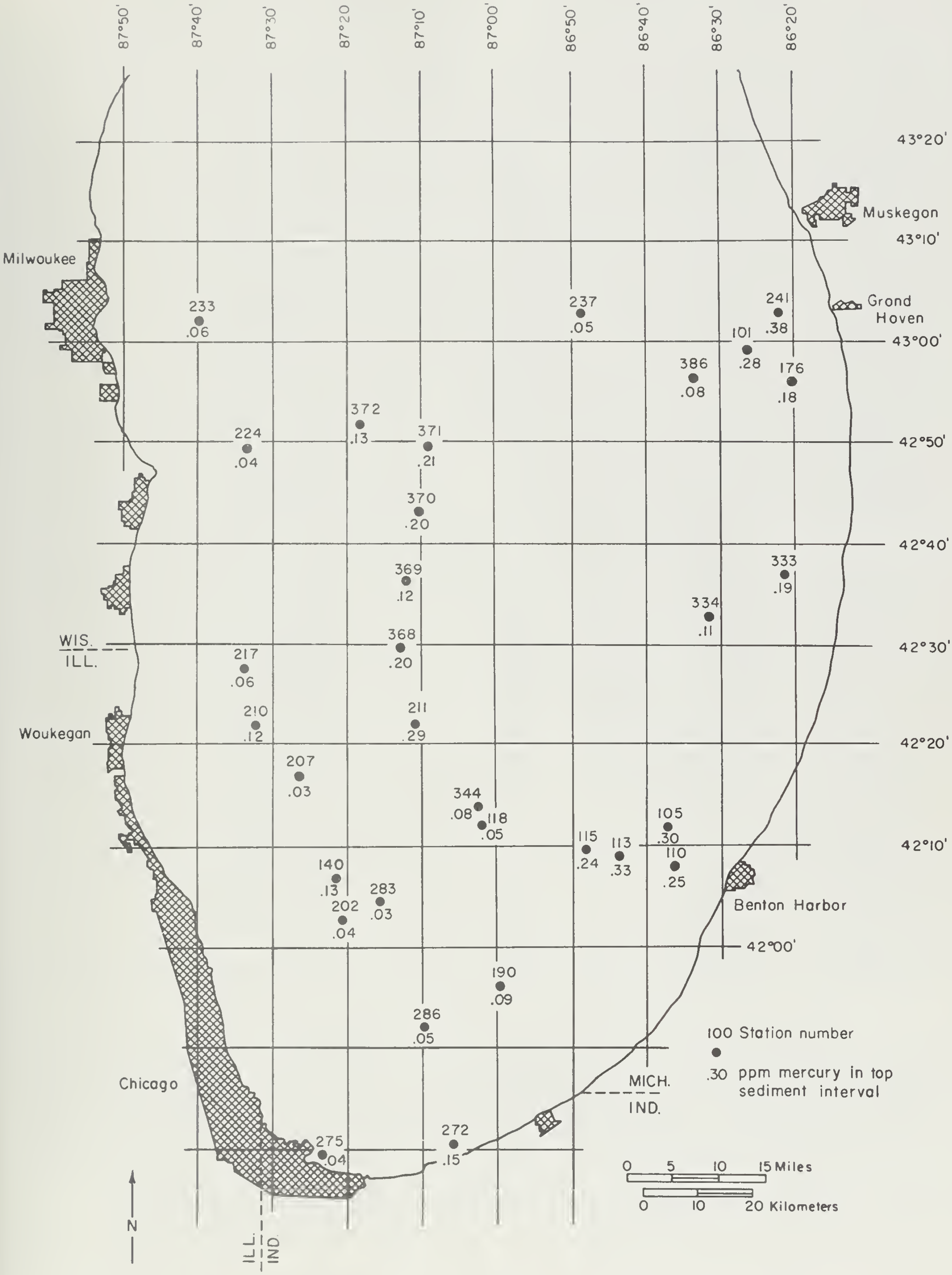

Fig. 1 - Sample locations and mercury content of the top interval of sediment at each location.

TABLE 1—TYPE OF SAMPLE AND LOCATION OF SAMPLING STATIONS

Station no.	Type of sample	Depth of water (ft)	Latitude	Longitude
101	3-in. core	250	42°59.2'N	86°25.9'W
105	3-in. core	168	42°13.8'N	86°37.0'W
110	3-in. core	102	42°8.0'N	86°36.5'W
113	3-in. core	230	42°9.0'N	86°43.8'W
115	3-in. core	276	42°9.8'N	86°48.1'W
118	2-in. core	312	42°12.0'N	87°1.8'W
140	3-in. core	200	42°6.8'N	87°21.4'W
176	3-in. core	175	42°56.2'N	86°20.4'W
190	3-in. core	188	41°56.3'N	86°59.6'W
202	3-in. core	175	42°2.7'N	87°20.4'W
207	3-in. core	265	42°17.0'N	87°26.2'W
210	3-in. core	260	42°21.8'N	87°32.1'W
211	3-in. core	387	42°21.8'N	87°10.8'W
217	3-in. core	258	42°27.5'N	87°33.6'W
224	3-in. core	239	42°49.2'N	87°33.2'W
233	3-in. core	210	43°1.8'N	87°39.5'W
237	3-in. core	323	43°2.7'N	86°49.0'W
241	3-in. core	212	43°3.0'N	86°22.3'W
272	Grab sample	54	41°40.5'N	87°6.2'W
275	Grab sample	36	41°39.4'N	87°23.5'W
283	Grab sample	207	42°4.4'N	87°15.6'W
286	Grab sample	125	41°52.1'N	87°9.6'W
333	3-in. core	180	42°37.3'N	86°21.4'W
334	3-in. core	250	42°33.0'N	86°31.9'W
344	3-in. core	324	42°13.8'N	87°2.4'W
368	3-in. core	451	42°29.5'N	87°12.8'W
369	3-in. core	480	42°36.4'N	87°11.8'W
370	3-in. core	491	42°42.9'N	87°10.2'W
371	3-in. core	481	42°49.3'N	87°9.0'W
372	3-in. core	398	42°51.8'N	87°18.0'W
386	3-in. core	300	42°56.5'N	86°33.5'W

Illinois Advanced TRIGA reactor for 110 minutes in a thermal neutron flux of 1.4×10^{12} neutrons cm^{-2} sec^{-1}. A standard irradiated along with the samples was an acidified mercuric nitrate solution containing 10.3 mg Hg^{++} per ml, which was sealed in a similar half-dram polyethylene vial. The samples and standard were rotated at 1 rpm in the reactor to ensure equal neutron flux.

Following irradiation, the samples were allowed to decay for 1 or 2 days. Each sample was then transferred to a 4-inch by 1/2-inch porcelain

combustion boat, which previously had been treated with a nonirradiated carrier solution containing 2.0 mg Hg^{++}, and then air dried. The irradiated sediment in the boat was completely covered with commercial-grade iron filings (about 5 to 7 g) that had been washed with water, alcohol, and ether to remove grease. The boat was then put into a Vycor tube, 26 mm in diameter and 41 cm long, and connected via a ground glass joint to two vacuum traps in succession. The Vycor tube was put into a tube furnace, which was heated to 800° C for about 20 minutes. A moderate flow of nitrogen was maintained through the tube, and the mercury vapor released was carried over into the traps, each of which contained 3 ml (30 mg Hg^{++}) of hold-back mercury carrier solution, 20 ml of sodium acetate—acetic acid buffer of pH 3.25, 40 ml of saturated bromine water, and deionized water to 100 ml total volume. The collecting solution is a modification of that of Hinkle, Leong, and Ward (1966). The collecting solutions were transferred to a beaker and the apparatus rinsed thoroughly with 2N HCl.

As trace amounts of mercury are easily adsorbed onto glass (Hinkle and Learned, 1969), between experiments all glassware was thoroughly cleaned of grease and rinsed with chromic acid solution and deionized water.

A 0.10 ml portion of the irradiated standard was diluted with 4M HNO_3 to 100 ml to give a solution containing 10.3 ppm Hg^{++}. One ml of this solution was put into a combustion boat, which was then air dried for at least 2 hours. About 1 gm of inactive sediment was put into the boat, iron filings were added, and the heating carried out as described for the samples.

The sample and standard collecting solutions were next passed through a 1- by 30-cm ion-exchange column containing 3.5 cm of Dowex 1-X8 resin. The column was rinsed with 20 ml of deionized water, and the mercuric ion was eluted with 90 ml of 4M HNO_3 into a 100 ml lidded polystyrene vial.

Gamma Ray Counting

The vials were mounted directly above a 3- by 3-inch sodium iodide crystal connected to a Nuclear Chicago 400-channel analyzer. The 77.6 keV gamma ray of ^{197}Hg ($t_{\frac{1}{2}}$ = 65 hr) was used to compare the activities of the samples to that of the standard.

A reference standard, consisting of 1 ml of the diluted standard solution (10.3 ppm Hg^{++}), 6 ml of Hg^{++} carrier solution (60 mg Hg^{++}), and 4M HNO_3 to a volume of 90 ml in a polystyrene vial, also was counted.

Comparison of the activity of this "untreated" standard with that of the treated standard was used to check the radiochemical yield obtained. Yields averaged 53 percent, with a standard deviation of 8.4 percent.

Analysis of the United States Geological Survey standard rock W-1 by our method gave the result 0.25 ppm ± 0.05 ppm mercury. Fleischer (1965) quotes the average values of 0.17 ppm obtained by neutron activation analysis and 0.34 ppm mercury obtained by atomic absorption spectrophotometry on W-1.

It is estimated that the precision of the method used here for determination of mercury in sediments is ± 20 percent.

RESULTS AND CONCLUSIONS

The mercury concentration of each core or grab sample sediment interval analyzed and the corresponding data for the $< 2\ \mu$ clay size fraction, organic carbon, and total sulfur are given in table 2. The mercury content of the top sediment interval at each station is shown in figure 1.

The results of the mercury analyses indicate that at most of the sampling stations an accumulation of mercury occurs at the top of the core, the concentration gradually decreasing down to a depth of 10 to 15 cm or lower, where a base level of 0.03 to 0.06 ppm mercury is reached. Distribution versus depth patterns of a variety of cores are illustrated in figures 2 through 10.

Several surveys have been made of the mercury content of the earth's crustal rocks. As summarized by Wedepohl (1970), all averages obtained for the igneous rocks at the earth's surface lie in the range of 0.06 to 0.09 ppm mercury. General correlations of the mercury contents of lake sediments with those of surrounding geologic formations have not been made.

As indicated in figure 1, areas in which the top intervals of the cores have relatively high mercury contents are west of Benton Harbor, Michigan, southwest of Grand Haven, Michigan, and in the deep central part of the southern part of the lake (stations 211 and 368 through 372, with water depths 380 to 500 feet). Areas of high mercury content in the top sediment interval are also, in general, areas of high organic carbon content. The distribution for mercury is very similar to the distributions observed for several other trace elements (Shimp et al., 1971).

The correlation of the mercury and organic carbon contents of the top sediment layer of all the cores is good (fig. 11). There is also an apparent correlation of the mercury and total sulfur contents of the top sediment interval samples (fig. 12), but a plot (fig. 13) of mercury versus the $< 2\ \mu$ clay size fraction concentrations for the top interval of the cores shows scatter.

In areas where the mercury content of the sediments is low, i.e., along the southern and southwestern shorelines, the organic carbon, total sulfur, and $< 2\ \mu$ clay size fraction concentrations also tend to be low. In these sandy regions there is in most cores an accumulation of mercury in a lower interval in the core (e.g., core 202 at 30 to 35 cm; core 207 at 30 to 34 cm; core 224 at 40 to 45 cm; core 233 at 33 to 37 cm; core 237 at 40 to 45 cm). There is also a detectable enrichment of total sulfur in the same interval of these cores. The mercury here may be associated with the presence of metal sulfides.

TABLE 2—MERCURY, LESS THAN 2-MICRON CLAY SIZE FRACTION, ORGANIC CARBON, AND TOTAL SULFUR CONCENTRATIONS OF LAKE MICHIGAN SEDIMENT SAMPLES*

			% of oven-dried (110° C) sediment		
Station no.	Depth (cm)	Mercury (ppm)	< 2 μ clay	Organic carbon	Total sulfur
101	0 - 2	0.28, 0.27	69	3.58	0.08
	2 - 4	0.17	56	2.33	0.05
	4 - 9	0.11	34	1.82	0.13
	33 - 37	0.04, 0.05	31	1.12	0.12
105†	0 - 3	0.30	—	4.73	0.20
	3 - 6	0.36	28	2.66	0.14
	6 - 9	0.35	31	2.93	0.16
	26 - 30	0.08	27	1.05	0.06
	90 - 94	0.05	31	1.72	0.08
110	0 - 3	0.25	19	1.88	0.06
	3 - 6	0.19	17	1.27	0.15
	6 - 9	0.22	9	0.91	0.10
	9 - 14	0.18	13	0.71	0.14
	14 - 18	0.08	11	0.49	0.07
113†	0 - 2	0.33	48	3.54	0.21
	2 - 4	0.26	45	3.29	0.12
	4 - 7	0.20	32	2.37	0.04
	30 - 34	0.67, 0.88	29	1.27	0.14
	91 - 95	0.05	35	1.39	0.06
115	0 - 3	0.24	48	3.81	0.07
	3 - 6	0.09	43	2.25	0.04
	6 - 9	0.05	45	1.92	0.03
	38 - 44	0.04	44	1.73	0.08
	98 - 103	0.05	48	1.13	0.07
118	0 - 7	0.05	54	0.64	0.02
	7 - 16	0.04	67	0.62	0.02
	16 - 20	0.06	56	0.61	0.02
140	0 - 3	0.13	13	1.18	0.03
	3 - 6	0.06	12	0.39	0.01
176	0 - 4.5	0.18	28	2.21	0.15
	4.5 - 9	0.14	39	1.81	0.13
	9 - 13	0.08	31	1.20	0.08
	30 - 35	0.03	19	1.12	0.08
190	0 - 3.5	0.09	14	0.80	0.04
	3.5 - 7	0.07	13	1.07	0.04
	10 - 15	0.08	18	2.43	0.23
	30 - 35	0.07	31	1.50	0.09

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TABLE 2—Continued

			% of oven-dried (110° C) sediment		
Station no.	Depth (cm)	Mercury (ppm)	< 2 μ clay	Organic carbon	Total sulfur
202	0 - 2	0.04	19	0.63	0.01
	2 - 6	0.04	39	0.20	0.01
	9 - 13	0.05	47	0.49	0.01
	13 - 17	0.04	37	0.36	0.01
	30 - 35	0.11	30	0.28	0.03
207	0 - 3	0.03	19	1.01	0.01
	3 - 6	0.02	32	0.66	0.01
	10 - 14	0.03	46	1.26	0.01
	30 - 34	0.06	57	0.94	0.03
210	0 - 3	0.12	—	—	—
	3 - 6	0.07	—	—	—
	6 - 10	0.05	—	—	—
	10 - 14	0.03	—	—	—
	14 - 18	0.04	—	—	—
	18 - 22	0.05	—	—	—
	22 - 26	0.04	—	—	—
	33 - 37	0.05	—	—	—
	63 - 67	0.05	—	—	—
	89 - 92	0.04	—	—	—
211	0 - 3	0.29	73	4.64	0.12
	3 - 6	0.22	65	3.67	0.14
	10 - 15	0.13	62	3.61	0.10
	30 - 35	0.09	64	3.41	0.14
217	0 - 5	0.06	24	1.32	0.07
	5 - 10.5	0.06	30	1.43	0.23
	10.5 - 15.8	0.07	42	0.98	0.10
	30 - 35	0.07	45	1.40	0.03
224	0 - 5	0.04	7	0.51	0.01
	5 - 10	0.03	18	0.37	0.01
	10 - 15	0.04	12	0.01	< 0.01
	40 - 45	0.10	26	0.50	0.10
233	0 - 5	0.06	21	0.62	0.01
	5 - 9	0.03	37	1.33	0.02
	9 - 14	0.06	47	1.23	0.02
	33 - 37	0.09	40	0.90	0.06
237	0 - 5	0.05	25	0.86	0.01
	5 - 10	0.05	38	0.40	0.01
	10 - 15	0.05	38	0.30	0.01
	40 - 45	0.12	83	0.33	0.20

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TABLE 2—Continued

Station no.	Depth (cm)	Mercury (ppm)	% of oven-dried (110° C) sediment		
			< 2 μ clay	Organic carbon	Total sulfur
241	0 - 4	0.38	19	2.21	0.09
	4 - 7	0.28	21	1.62	0.15
	7 - 10	0.12	25	1.76	0.16
	10 - 14	0.03	23	0.85	0.06
	14 - 18	0.06	19	1.22	0.09
272	0 - 1	0.15	18	—	—
	1 - 3	0.11	11	—	—
	3 - 5	0.02	8	—	—
	5 - 7	0.07	7	—	—
275	0 - 3	0.04	6	—	0.07
283	0 - 1	0.03	9	—	0.03
	1 - 3	0.02	18	—	0.02
	3 - 5	0.03	20	—	0.02
	5 - 7	0.03	27	—	0.02
	7 - 9	0.02	32	—	0.03
286	0 - 1	0.05	38	—	0.10
	1 - 3	0.08	58	—	0.16
	3 - 5	0.05	62	—	0.21
333	0 - 5	0.19	25	1.61	0.13
	5 - 9	0.24	22	1.33	0.09
	9 - 13	0.15	26	1.62	0.09
	30 - 35	0.04	20	0.87	0.07
334	0 - 5	0.11	58	2.47	0.09
	5 - 9	0.04	44	0.52	0.01
	9 - 13	0.10	43	2.29	0.08
	30 - 35	0.06	42	2.06	0.08
344	0 - 5	0.08	77	0.61	0.01
	5 - 9	0.10	86	2.23	0.09
	9 - 13	0.07	78	0.30	0.01
	30 - 35	0.05	86	0.60	0.01
368	0 - 3	0.20	33	3.96	0.04
	3 - 7	0.12	47	3.36	0.04
	10 - 15	0.06	55	2.28	0.22
	30 - 35	0.06	68	3.21	0.20
	71 - 76	0.05	66	2.50	0.24

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TABLE 2—Concluded

			% of oven-dried (110° C) sediment		
Station no.	Depth (cm)	Mercury (ppm)	< 2 μ clay	Organic carbon	Total sulfur
369	0 - 2	0.12	28	3.10	0.33
	2 - 6	0.08	64	2.78	0.23
	10 - 15	0.08	88	3.29	0.18
	30 - 35	0.05	71	2.70	0.36
370	0 - 3	0.20	15	4.71	0.06
	3 - 6	0.15	17	4.21	0.07
	6 - 10	0.11	22	3.70	0.17
	10 - 15	0.05	55	3.15	0.21
	30 - 35	≤ 0.04[‡]	57	3.40	0.25
371	0 - 3	0.22, 0.21	44	5.28	0.03
	5 - 7	0.17, 0.19	56	3.97	0.04
	9 - 15	0.03	68	3.02	0.08
	31 - 36	0.05	62	3.50	0.05
372	0 - 4	0.13	57	3.22	0.04
	4 - 7	0.07	69	1.71	0.02
	10 - 15	0.05	67	1.19	0.01
	30 - 35	0.07	68	0.99	0.08
386	0 - 5	0.08	47	1.61	0.04
	5 - 9	0.04	46	2.04	0.17
	9 - 13	0.07	54	2.28	0.57
	30 - 35	0.07	41	2.18	0.28

[*] Organic carbon analyses were done by L. R. Camp and C. W. Beeler, < 2 μ clay size fraction determinations by W. A. White, and total sulfur analyses by L. R. Camp. Complete descriptions of the methods of analysis and some of the results are given in Environmental Geology Notes 32 and 41.

[†] The < 2 μ clay size fraction values for cores 105 and 113 used in figures 7 and 9 of Environmental Geology Note 37 were not correctly listed in table 2 of that publication. The top sediment interval value of core 113 in figure 19 of that publication also was incorrect.

[‡] Bromine interference prevents accurate measurement.

ACKNOWLEDGMENTS

Acknowledgment is made to the TRIGA staff for their cooperation in performing the irradiations, to Dr. Harry V. Leland of the Civil Engineering Department of the University of Illinois for the major part he played in the selection of sampling stations and acquisition of the samples analyzed, and to the Great Lakes Research Division, Ann Arbor, Michigan, for ship time and facilities.

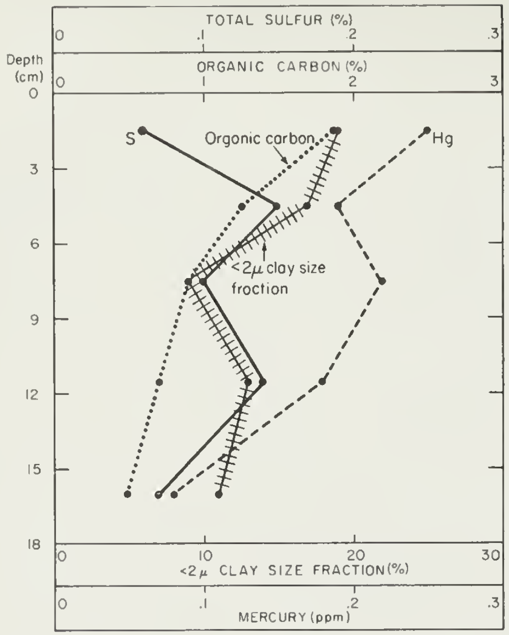


Fig. 2 - Distribution of mercury, organic carbon, total sulfur, and < 2 μ clay size fraction in Lake Michigan core 110.

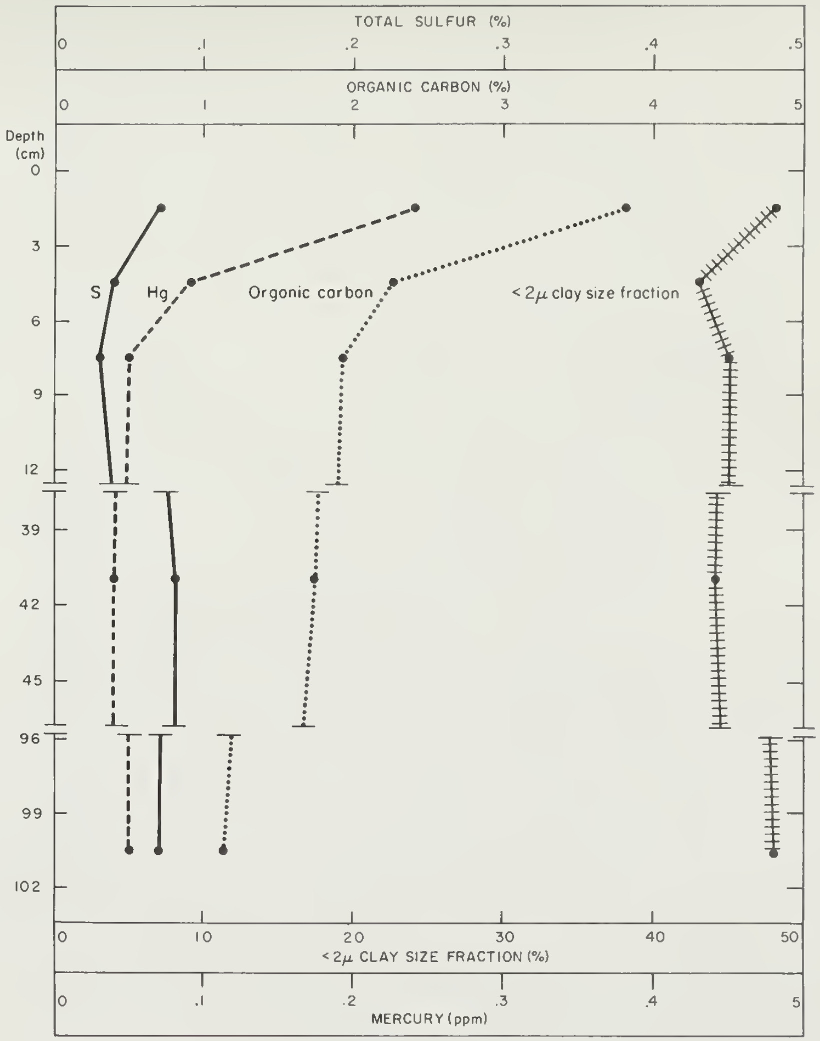


Fig. 3 - Distribution of mercury, organic carbon, total sulfur, and < 2 μ clay size fraction in Lake Michigan core 115.

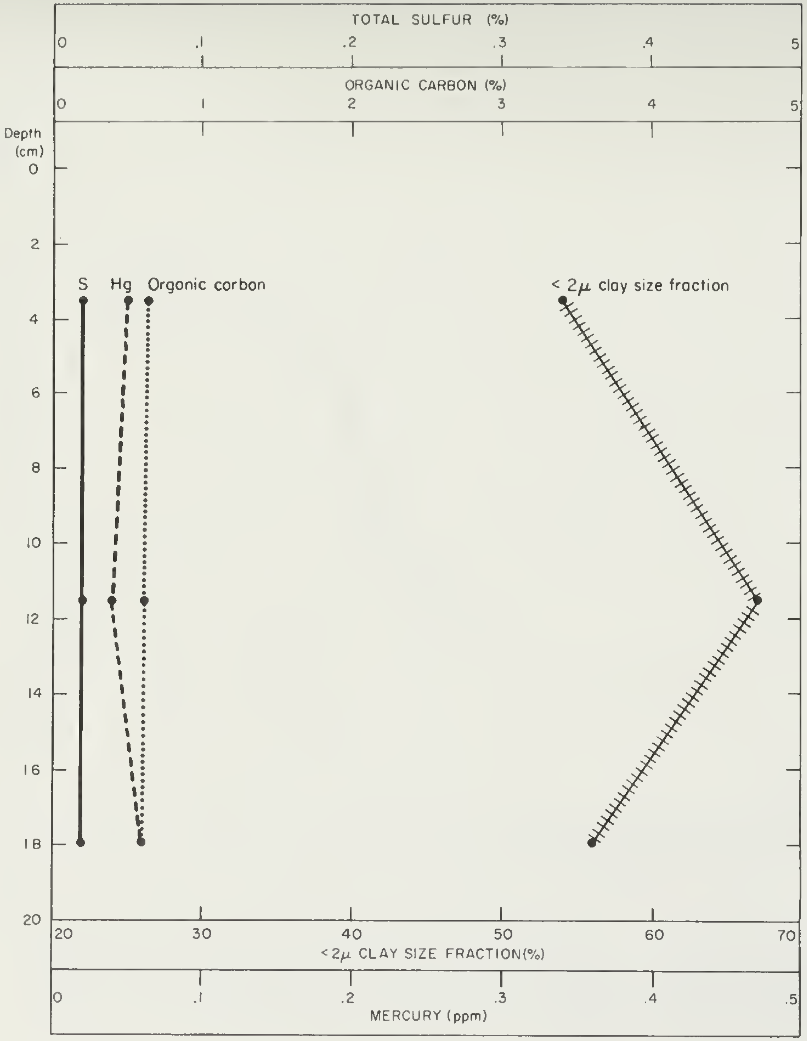


Fig. 4 - Distribution of mercury, organic carbon, total sulfur, and < 2 μ clay size fraction in Lake Michigan core 118.

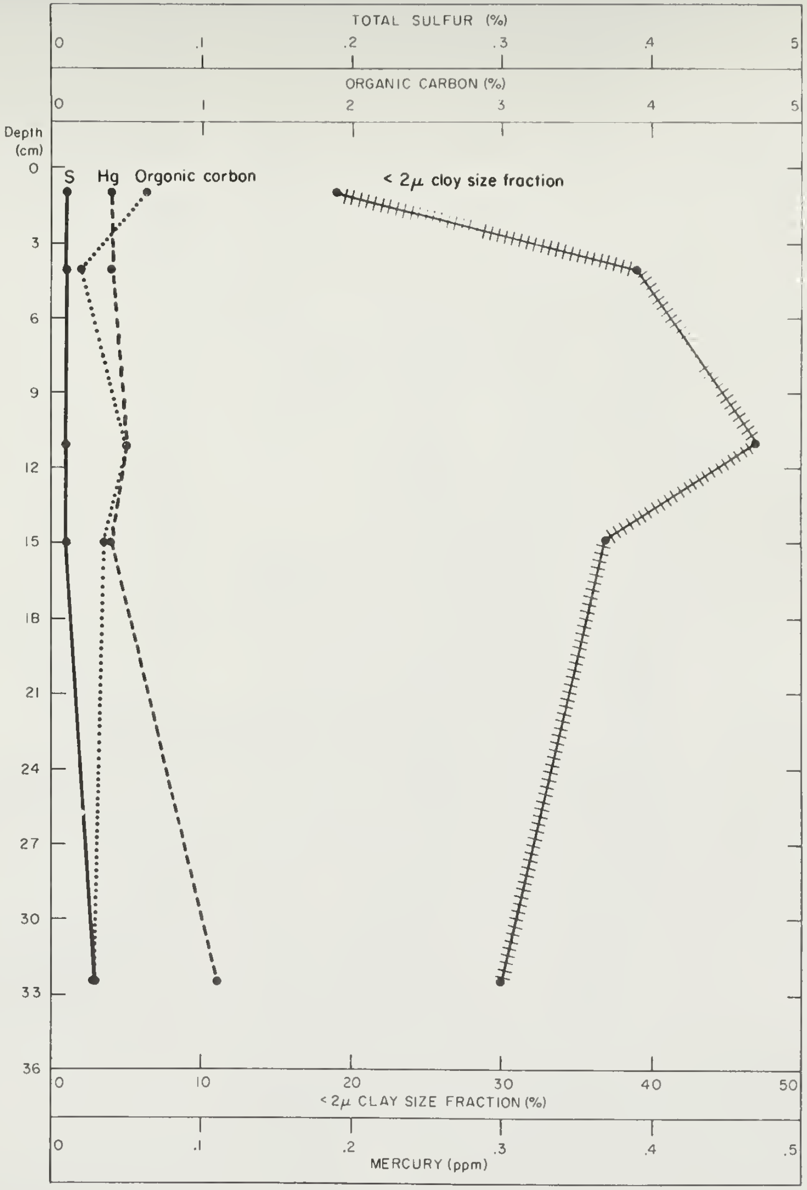


Fig. 5 - Distribution of mercury, organic carbon, total sulfur, and < 2 μ clay size fraction in Lake Michigan core 202.

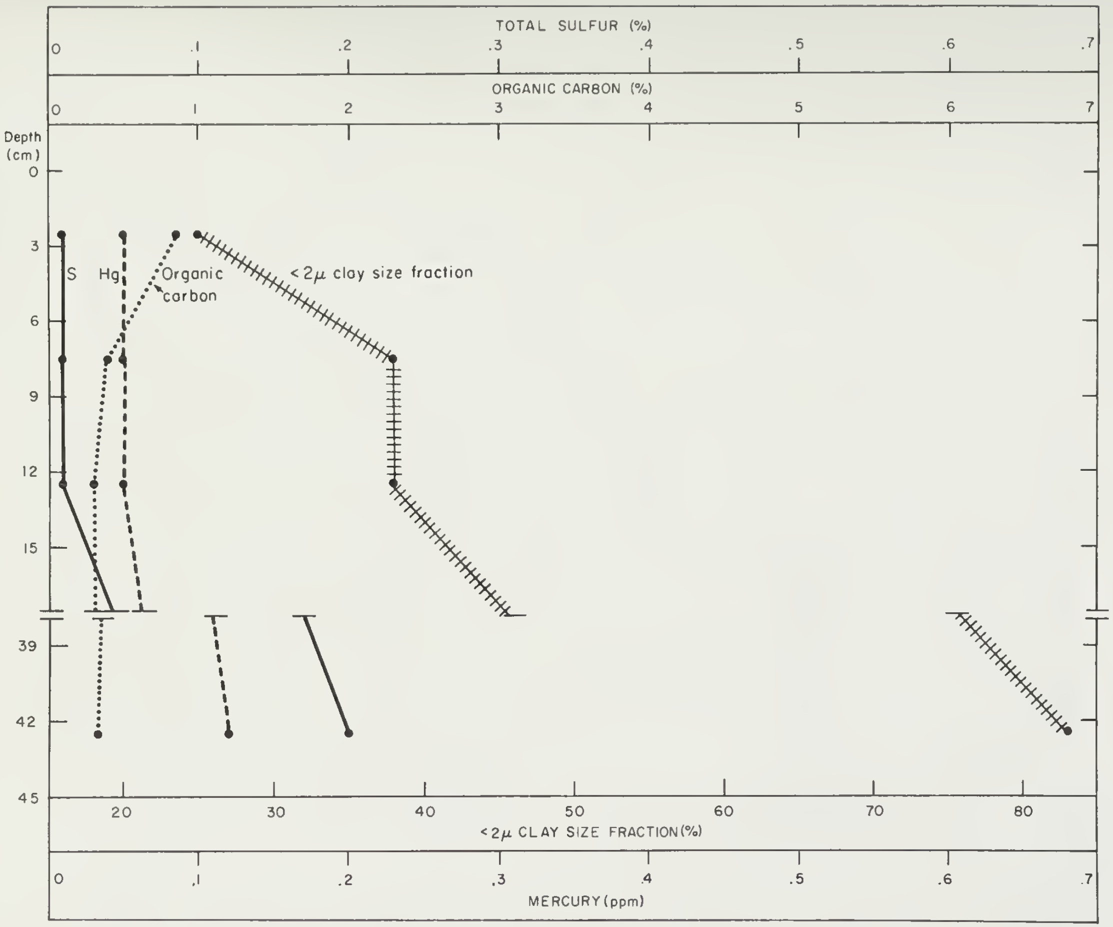


Fig. 6 - Distribution of mercury, organic carbon, total sulfur, and < 2 μ clay size fraction in Lake Michigan core 237.

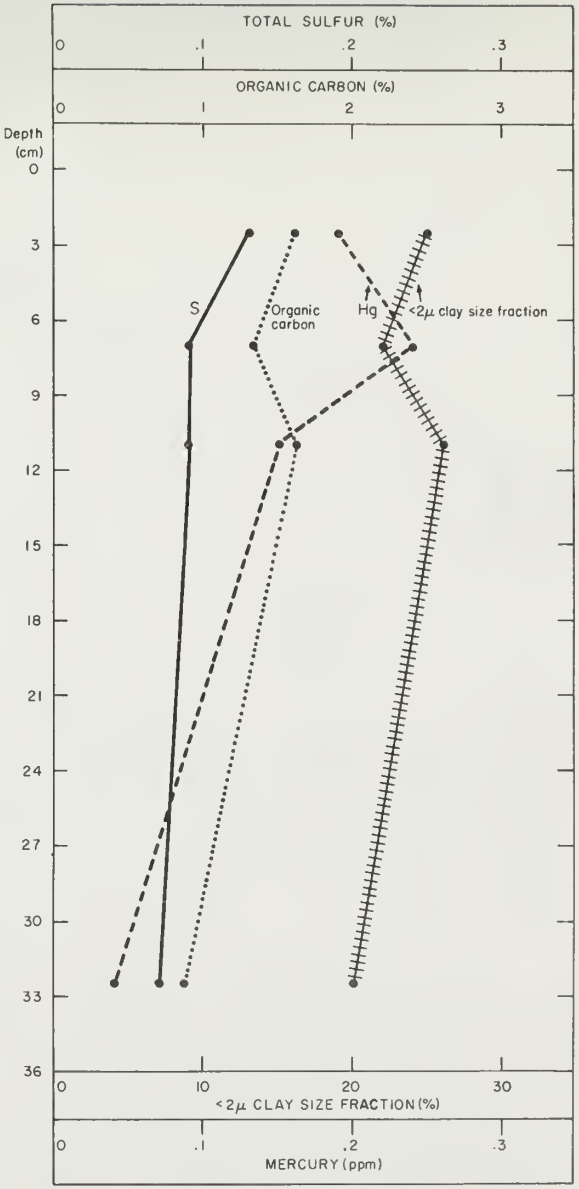


Fig. 7 - Distribution of mercury, organic carbon, total sulfur, and < 2 μ clay size fraction in Lake Michigan core 333.

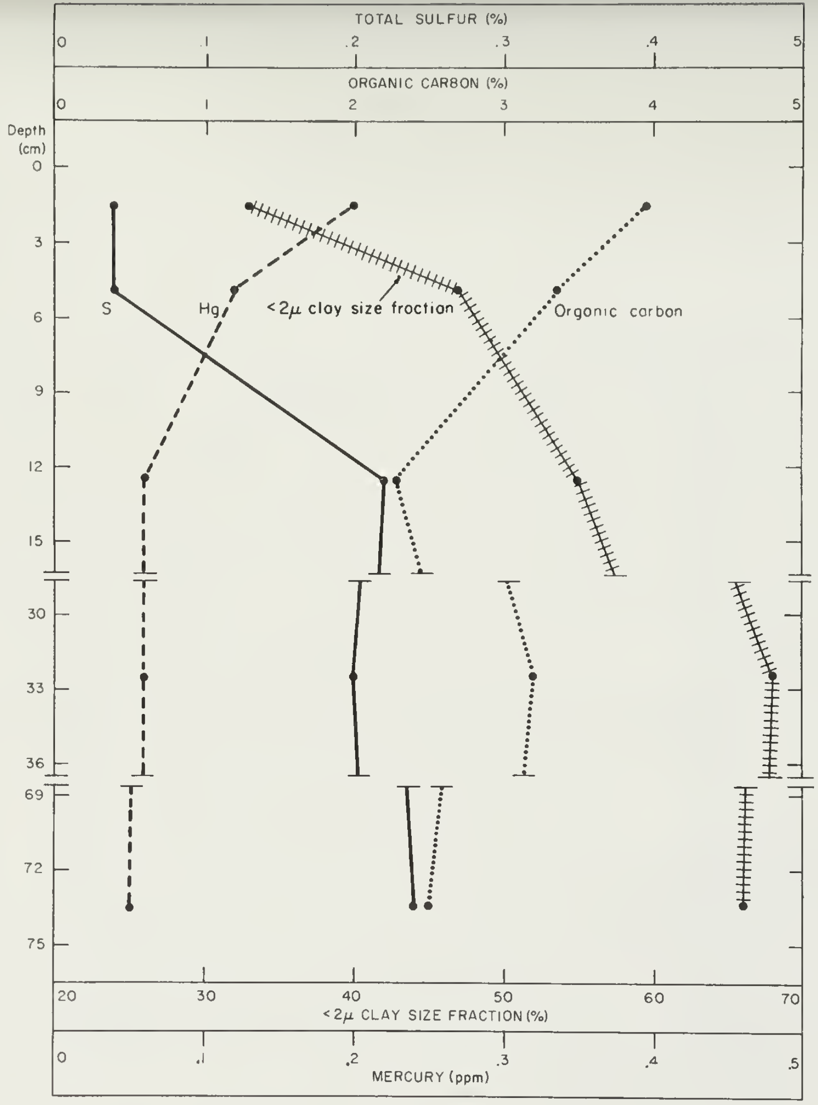


Fig. 8 - Distribution of mercury, organic carbon, total sulfur, and < 2 μ clay size fraction in Lake Michigan core 368.

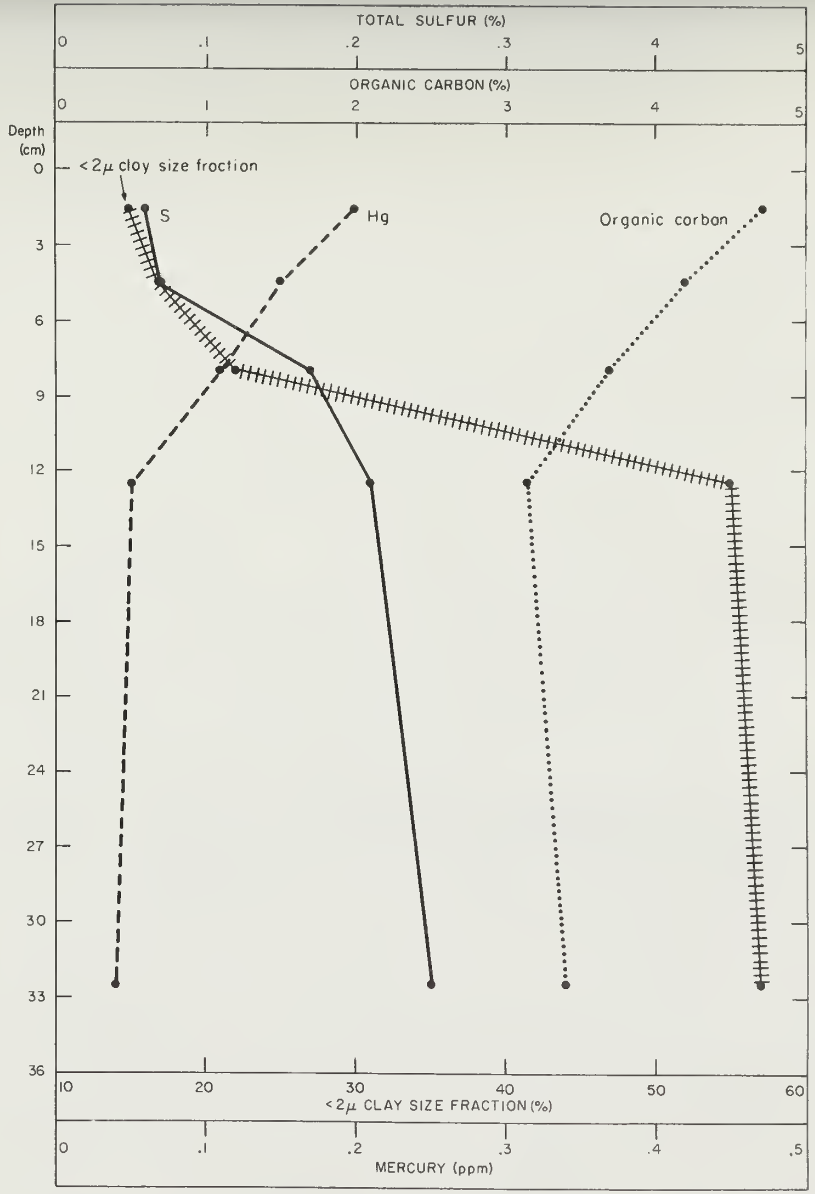
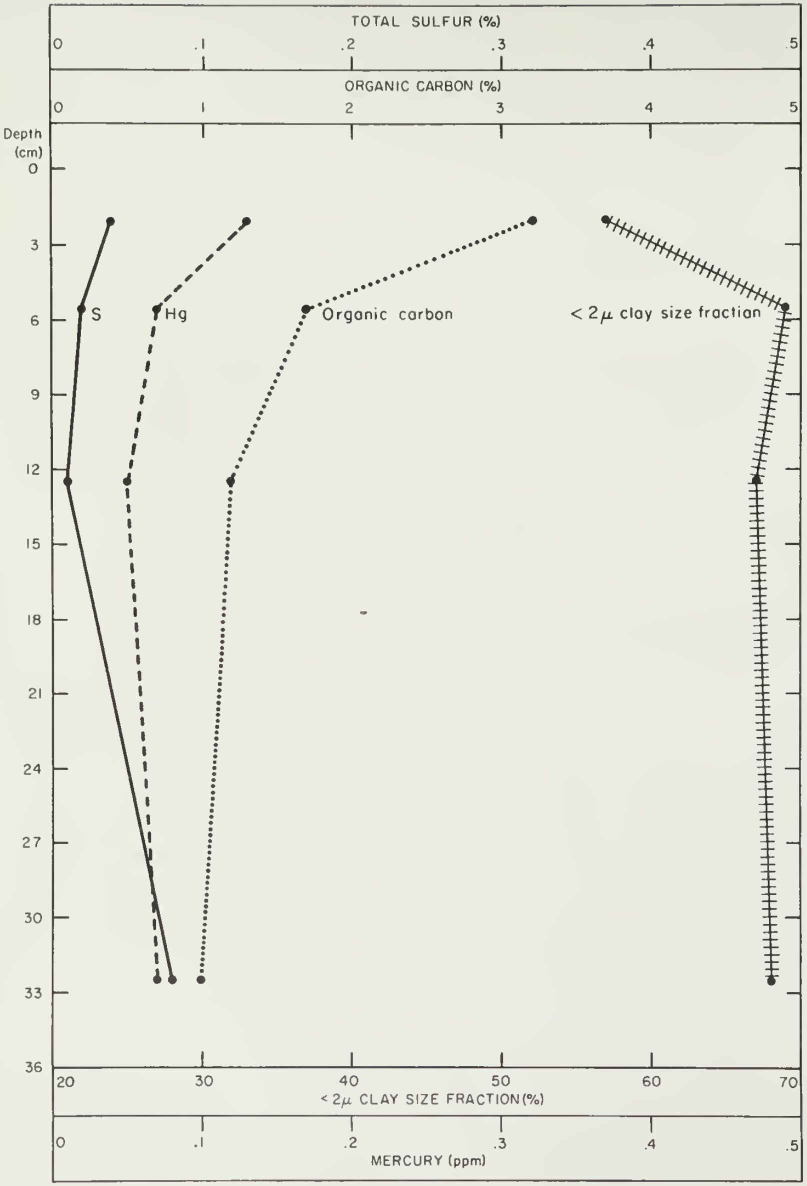


Fig. 9 - Distribution of mercury, organic carbon, total sulfur, and < 2 μ clay size fraction in Lake Michgian core 370.



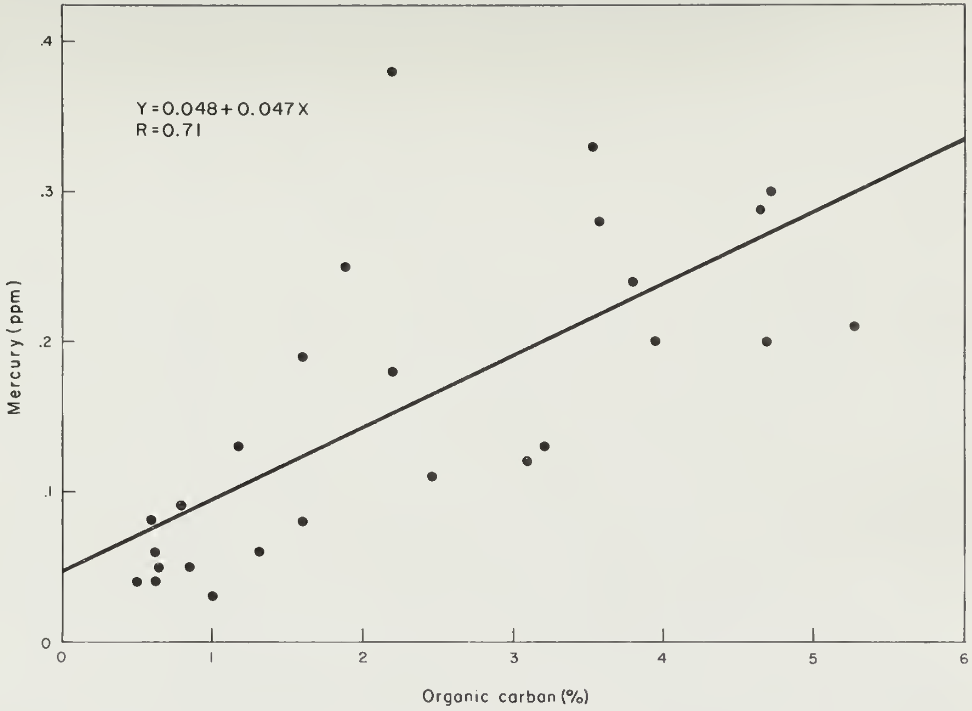


Fig. 11 - Correlation of mercury and organic carbon concentrations in the top interval of 26 core and grab samples.

Fig. 10 - Distribution of mercury, organic carbon, total sulfur, and < 2 μ clay size fraction in Lake Michigan core 372.

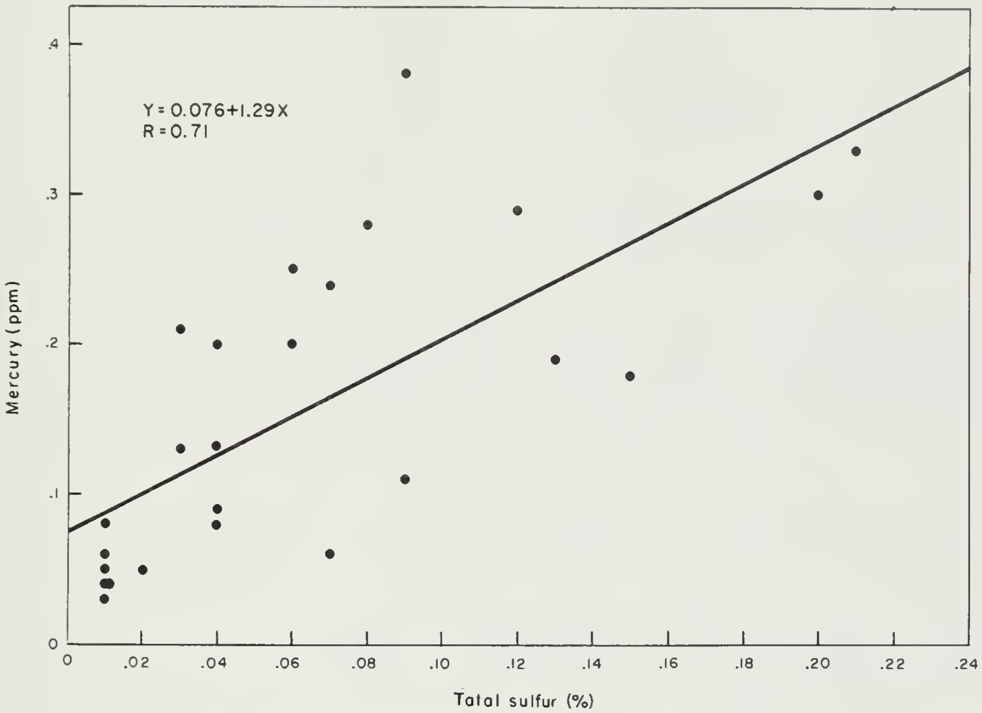


Fig. 12 - Correlation of mercury and total sulfur concentrations in the top interval of 25 core and grab samples.

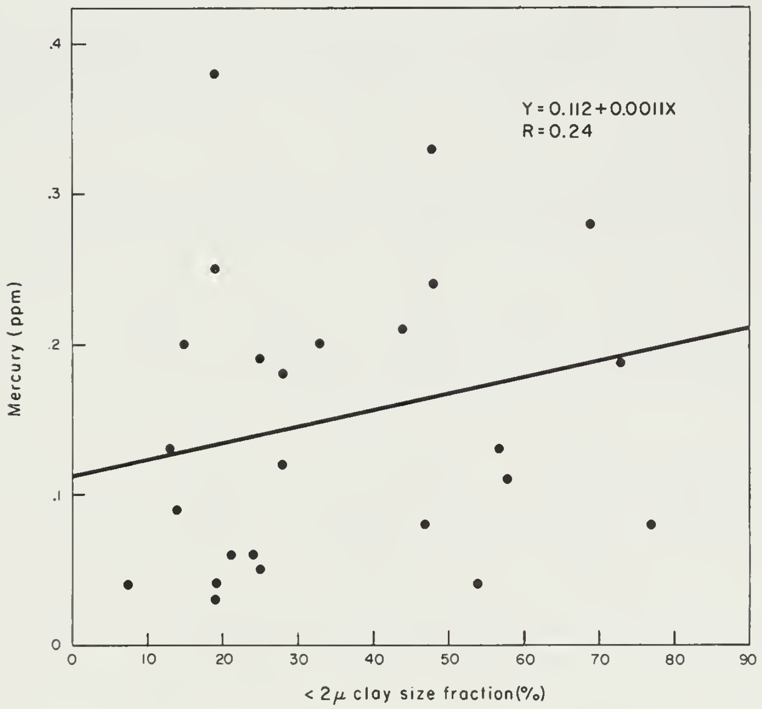


Fig. 13 - Correlation of mercury and < 2 µ clay size fraction concentrations in the top interval of 25 core and grab samples.

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